

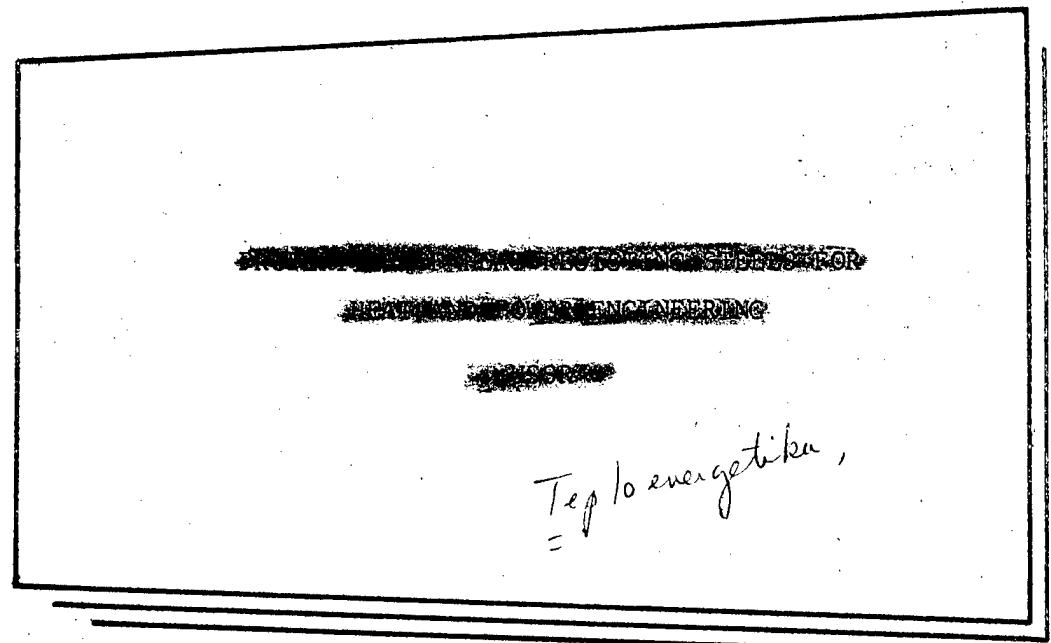
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RESTORING THE INITIAL HEAT-RESISTING PROPERTIES  
OF MATERIALS BY INTERMEDIATE HEAT TREATING

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61148

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Results are presented of a study of  
the degree of damage at various stages  
of creep of specimens of a steel of  
the austenitic class.

Numerous studies of the mechanism of the plastic  
deformation and the fracture of materials under conditions  
of creep at high temperatures have shown that the predominant  
roles in the phenomenon of stress rupture are played by the  
diffusion processes. In several studies, Ref 1 for example,  
the reversibility of the stress-rupture process in the early  
stages of its development has been shown.

A significant increase in the service life of ma-terials (Ref 2) has been achieved by an intermediate heat treatment. In this case it was noted that at relatively low stresses the intermediate heat treatments could increase the service life of the material by several times. At the higher stresses the susceptibility appears to a lesser degree and the possibility of slowing the stress rupture process is reduced.

The recovery of the properties after long-term operation of the material of turbine blades was shown by Glikman in his study of the variation of the damping decrement (Ref 4).

The intermediate heat treatment, in addition to slowing of the stress-rupture process, can restore the initial structure and the phase composition, aids in the equalization of properties between individual grains and the redistribution of the microstresses with the elimination of the peaks in the areas of stress concentration. All this can lead to an increase of the service life of the parts or to an increase of the working loads for the same duration of operation.

However the question has still not been resolved of to what stage the development of the stress-rupture process is reversible and to what degree we can "heal" the fracture centers by the intermediate heat treatment. The present article presents the results of an experimental verification in laboratory conditions of the possibility of healing damage

accumulated in the material at various stages of the fracturing under creep conditions.

As the object of the study we used the metal of the working section of specimens tested in creep and stress-rupture of several high-temperature steels: the EI-257, EI-1T steels and a nickel alloy.)

Table 1 presents the test conditions for these specimens. We see that the test duration varied from several hundred to several thousand hours, i.e. the testing was interrupted at various stages of the stress-rupture testing. The extent of damage was evaluated on the basis of the change of the density of the metal. The magnitude of the density was determined by the method of hydrostatic weighing. One specimen of each pair from the EI-257 steel after the creep test was subjected to heat treatment (heated to 800°C, soak for 5 hours, furnace cooled) to heal the accumulated damage. The error in the measurement of the density did not exceed 0.001 which corresponded to 5-10% of the measured values.

Table 1

№ группы a	Марка стали b	Температура испытания, °С c	Напряжение σ, кг/мм <sup>2</sup> d	Длительность испытания, ℓ e	Число образ- цов m	Примечание n
L Первая группа	ЭИ-257 f	580	24	200	2	
		580	24	500	2	
		580	21	1 000	2	
		580	24	1 500	2	
		580	24	2 000	2	
		580	24	2 500	2	
C Вторая группа	ЭИ-257 f	580	24	3 500	2	
		580	26	100	8	
		580	26	300	8	
		580	26	500	8	
D Третья группа	Никелевый сплав h ЭИ-1T	750	30	140	1	Образец разорвал- ся
		750	25	585	1	
		600	20	1 846	1	
		600	22	979	1	
I Сталь 45	i	600	24	851	1	То же
		20	-	-	4	Кратковременный разрыв

a - group No; b - first group; c - second group; d - third group; e - steel grade; f - EI-257; g - nickel alloy; h - EI-1T; i - steel; j - test temperature; k - stress in kg/mm<sup>2</sup>; l - test duration in hours; m - number of specimens; n - remarks; o - specimen failed; p - same; q - short-term tension

In one group of the studies the accuracy of the determination of the density was increased by testing of parallel samples. In another group an improvement of the reliability of the estimate of the degree of damage was achieved by the use of the Machlin technique, (Ref 1). The exposure of the pores and microcracks obtained under the creep conditions was accomplished by means of active deformation at lower temperatures. For this purpose all the specimens were subjected to a uniform elongation of 10% at normal temperature. Such a deformation should aid in the exposure of the pores and microcracks accumulated under the creep conditions. If the heat treatment had healed over the fracture centers, then during the cold deformation no discontinuities should develop in the metal.

Fig 1 presents a plot of the variation of the density of the metal after creep (curve 1) and after the subsequent heat treatment (curve 2) for the specimens of the first group ( $\sigma = 24 \text{ kg/mm}^2$ ).

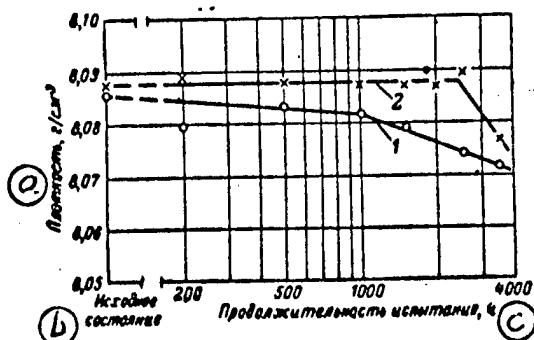


Fig 1. Variation of the density of EI-257 steel during creep as a function of test duration

$$\Theta = 580^\circ\text{C}, \sigma = \text{kg/mm}^2$$

1 - after creep test the specimen was work hardened by 10% elongation in the cold condition; 2 - after creep testing the sample was heat treated by heating to  $800^\circ\text{C}$ , soak for 5 hours, then work hardened in the cold condition by 10%  
a - density,  $\text{g/cm}^3$ ; b - initial condition; c - test duration, hours

We note the following from Fig 1. The damage of the metal starts in the first stage of creep; after 200 and 500 hours of testing there is a reduction of the density. With

an increase of the testing time there is a noticeable acceleration of the damage process; after 1000 hours the rate of decrease of the density goes up. The major portion of the defects are subject to healing with heat treatment if the time to failure is not more than half used up, up to 2500 hours the density is practically completely restored; with longer test duration the magnitude of the density increases insignificantly, i.e. only the small failure centers are subject to healing. In the second group the stress rupture test was conducted at a higher stress ( $\sigma = 26 \text{ kg/mm}^2$ ). Considering the static nature of the stress-rupture test and the associated scatter of the experimental data, we determined the mean time to failure from the results of numerous repeated tests.

After evaluating in this fashion the expected time to failure ( $\bar{\theta}_1 \approx 500$  hours) we conducted tests using the same conditions in three series with eight samples in each (Table 1). The duration of the testing of the first series was 20% of the mean life:  $\theta_1 = 0.2 \bar{\theta}_k$ . In the second series

$\theta_2 = 0.6 \bar{\theta}_k$ . The duration of the tests of the third series of samples was equal to the mean life ( $\theta_3 = \bar{\theta}_k$ ), i.e. the failure process reached the final stage.

[After the stress rupture tests half of the samples of each series were subjected to heat treatment for the purpose of healing the fracture centers. (austenitizing at 1100°C, soak for 30 min).] In this group the estimate of the damage was made on the basis of the results of the determination of the mechanical properties of the steel at normal temperature.] Table 2 presents the results of the test in short-term tension of the samples tested for creep. Table 3 presents analogous results of the test in short-term tension of the samples which were subjected to heat treatment after the creep testing.

We see from the tables that the damage in the creep conditions has the greatest effect on the plasticity characteristics ( $\sigma$  and  $\psi$ ) and on the true resistance to fracture ( $S_k$ ). If we judge on the basis of the variation of  $\delta$ ,  $\psi$  and  $S_k$  then we can note that for a test duration of 300 hours, i.e. about 40-50% of the service life, the healing completely liquidates the creep damage. Increase of the test duration leads to the formation of stable microcracks and the failure process becomes irreversible.

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Table 2. Mechanical properties of EI-257 at 200 after stress rupture testing at 580C and 26 kg/mm<sup>2</sup>

Продолжительность испытания на длительную прочность, $\text{ч}$ ( <i>a</i> )	Предел ползучести $\sigma_s$ , $\text{kgc/mm}^2$ ( <i>b</i> )	Предел прочности $\sigma_b$ , $\text{kgc/mm}^2$ ( <i>c</i> )	Истинное нормальное конечное напряжение $S_k$ , $\text{kgc/mm}^2$ ( <i>d</i> )	Относительное удлинение $\delta, \%$ ( <i>e</i> )	Относительное сужение $\phi, \%$ ( <i>f</i> )
100	39,6 39,9	63,9 62,6	176,6 153,5	50,6 56,0	60,9 65,0
Среднее <i>g</i> 300	39,8	62,2	166,2	57,8	67,0
	41,9	64,4	172,2	59,6	72,1
	39,9	61,7	132,1	36,8	51,2
Среднее 500	40,0	63,0	152,2	48,2	61,7
	44,3	68,8	98,5	25,2	31,2
	44,6	69,5	113,4	32,4	40,9
Среднее	41,4	69,2	106,0	28,8	36,0

a - duration of stress rupture test; b - creep limit; c - ultimate strength; d - true normal final stress; e - relative elongation; f - relative reduction; g - average

Table 3. Mechanical properties of EI-257 steel at 200 after stress rupture testing at 580C and 26 kg/mm<sup>2</sup> and a restoring heat treatment at 1100C for 30 minutes

Продолжительность испытания на длительную прочность, $\text{ч}$ ( <i>a</i> )	Предел текучести $\sigma_s$ , $\text{kgc/mm}^2$ ( <i>b</i> )	Предел прочности $\sigma_b$ , $\text{kgc/mm}^2$ ( <i>c</i> )	Истинное нормальное конечное напряжение $S_k$ , $\text{kgc/mm}^2$ ( <i>d</i> )	Относительное удлинение $\delta, \%$ ( <i>e</i> )	Относительное сужение $\phi, \%$ ( <i>f</i> )
100	20,8 20,5	54,3 55,4	193,3 194,7	74,4 76,4	76,1 78,6
Среднее <i>g</i> 300	20,6	54,8	194,0	75,4	77,4
	20,3	55,3	201,0	82,4	79,8
	19,5	55,2	199,1	71,6	76,8
	19,8	53,8	185,8	79,2	77,1
Среднее 500	19,9	51,8	192,5	77,7	77,9
	18,8	54,8	181,6	70,8	66,5
	20,1	52,4	117,0	57,2	56,1
Среднее	19,5	53,6	134,3	64,0	61,3

a - duration of stress rupture test; b - creep limit; c - ultimate strength; d - true normal final stress; e - relative elongation; f - relative reduction; g - average

Thus both groups of tests indicated that the intermediate heat treatment permits the liquidation of the major portion of the failure centers if not more than half of the creep service life has been used up.

In the third group we used as test specimens the remains of the samples which had been tested to failure in creep or in short-term tension. One half of these samples were subjected to healing by heat treatment. The estimate of the degree of damage was made by the measurement of the density. The density of the metal of each half of the sample was determined from the results of triple measurements. After the first measurement, a portion of the metal was removed by a cutter (lathe) from the end opposite the location of the failure. Then the density of the reduced metal sample was measured. This operation was repeated several times. The first measurement essentially determined the density of the portion of the metal of the working part of the samples located far from the fracture. As the samples were reduced in size by removal of metal from one end, the tested portion approached the fracture location, the zone of maximum damage. The final measurement of the density determined the density of a small volume of metal in which the damage is at a maximum. Thus we established the variation of the density as a function of the metal volume, i.e. as a function of the degree of damage in the stress rupture test. 6p.17

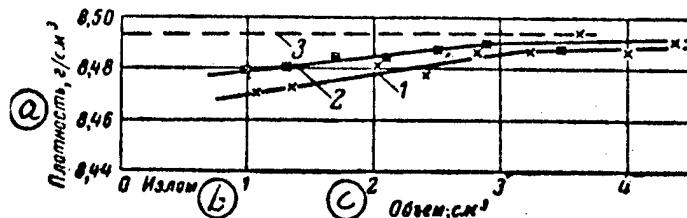


Fig 2. Variation of the density of the forging alloy TsZH-6 after stress rupture ( $\theta = 750^\circ C$ ,  $\sigma = 30 \text{ kg/mm}^2$ ,  $\theta_k = 140 \text{ hours}$ ,  $\epsilon_k = 9.0\%$ ,  $\psi = 19.0\%$ )

1 - after testing; 2 - after testing and heat treatment at  $1200^\circ C$  for 1.5 hours, annealed at  $750^\circ C$  for 20 hours; 3 - initial condition; a - density in  $\text{g/cm}^3$ ; b - fracture; c - volume,  $\text{cm}^{-3}$

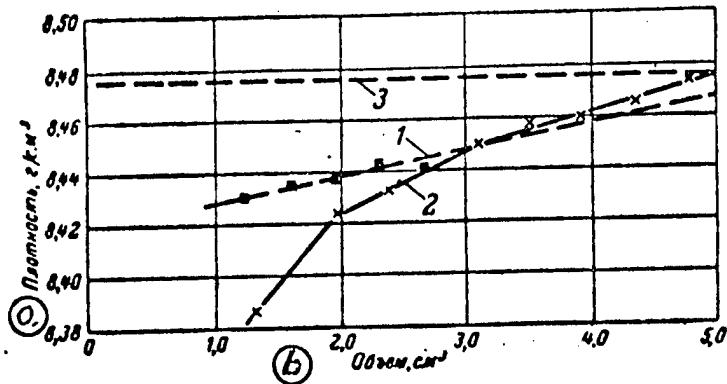


Fig 3. Variation of the density of the forged alloy TsZh-6 after stress rupture ( $\theta = 750^\circ C$ ,  $\sigma = 25 \text{ kg/mm}^2$ ,  $\theta_k = 585 \text{ hours}$ ,  $\varepsilon_k = 9.2\%$ ,  $\psi_k = 22.8\%$ )

1 - after testing; 2 - after testing and heat treatment at  $1200^\circ C$  for 1.5 hours, anneal at  $750^\circ C$  for 20 hours; 3 - initial condition; a - density,  $\text{g/cm}^3$ , b - volume,  $\text{cm}^{-3}$

Figs 2 and 3 present plots of the variation of the density of two samples of the nickel alloy. Fig 4 shows the variation of the density in creep conditions of one of the samples of the grade EI-1T steel (Table 1). First of all we should note that in both cases plastic deformations of similar magnitude, both overall ( $\varepsilon_k$ ) and local ( $\psi_k$ ) preceded the fracture.

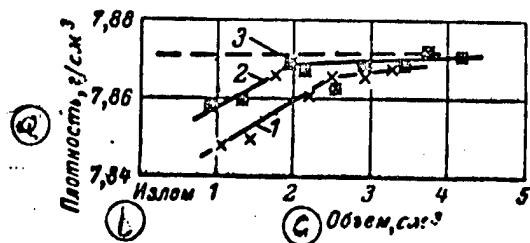


Fig 4. Variation of the density of the EI-1T alloy after stress rupture ( $\theta = 600^\circ C$ ,  $\sigma = 20 \text{ kg/mm}^2$ ,  $\theta_k = 1846 \text{ hours}$ )

1 - after testing; 2 - after testing and heat treatment at  $1040-1100^\circ C$  for 30 min, anneal at  $800^\circ C$  for 10 hours; 3 - initial condition; a - density,  $\text{g/cm}^3$ ; b - fracture; c - volume

Analysis of the results of the study of the accumulation of defects in the zones adjacent to the fracture showed that during the first stage of creep the initiation and the growth of the defects takes place throughout the entire volume of the working portion of the samples. However in this stage the dimensions of the defects are small and they are subject to healing; the magnitude of the density reaches the initial level (Figs 2 and 4).

Increase of the time up to the point of failure leads to a noticeable localization of the failure processes in the volume of the working portion of the samples. Right in the region of the fracture there is noted a significant acceleration of the decrease of the density (Figs 3 and 4). In this zone there probably occurs growth of the defects, most of which attain a critical dimension and their healing becomes impossible; after the restoring heat treatment the density of the metal is restored only by 50%. Judging from the plots of Figs 3 and 4 the healing is effective only prior to the localization of the failure process.

Some additional information on the kinetics of the stress rupture is given by a comparison of the results of the study of the investigation of the samples of the first (Fig 1) and the third (Figs 3 and 4) groups. In the first group of the tests during the creep process there was no localization of the plastic deformation along the length of the working section noted in any of the samples (absence of a neck) and judging by the initial curves for the stress-rupture (Fig 5) the third stage of accelerated creep did not appear.

If we compare these data with the results of the healing (Fig 1) then we can note that a large number of the defects reach the critical dimension prior to the onset of the third stage of creep in the absence of the localization of the development of the plastic deformation. Actually, after 3500 hours of testing (Fig 1) there is observed a slight increase of the density after the heat treatment, i.e. a significant number of the defects exceeded the critical dimension.

It appeared of interest to study the possibility of healing the failure centers formed during the active deformation under conditions of normal temperatures, i.e. when the diffusion processes do not play any role in the initiation and the growth of the fracture centers. To answer this question we carried out similar investigations on the remains of the samples of the steel 45 after short-term tension fracture at normal temperature.

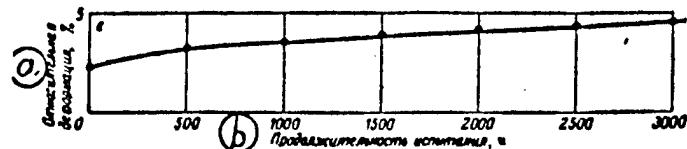


Fig 5. EI-257 steel. Initial stress rupture curve at  $\theta = 580^\circ\text{C}$ ,  $\sigma = 24 \text{ kg/mm}^2$ . a - relative deformation; b - test duration, hours

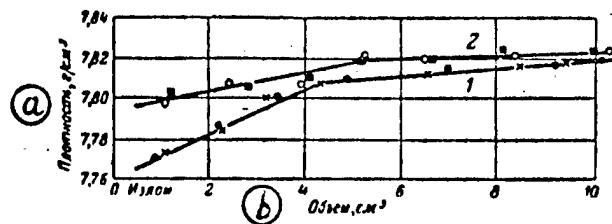


Fig 6. Variation of the density of steel 45 after short-term tension fracture  
 1 - after testing; 2 - after testing and heat treatment at  $800^\circ\text{C}$  for 5 hours  
 a - density,  $\text{g/cm}^3$ ; b - volume,  $\text{cm}^3$

Fig 6 is a plot of the tests of the two samples. The results of the measurement of the density of the metal of both samples practically coincided and this permitted drawing single curves for the variation of the density prior to and after heat treatment. Comparison of the graphs of Figs 4 and 6 shows that the variation of the density, and consequently the development of the fracture centers along the volume of the working portion during active deformation, does not differ in nature from the development of the defects in the samples during the creep testing. On the basis of these results we can draw the conclusion that defects formed by both the diffusion and the shear formation mechanisms are subject to healing.

[The results of the study made permit the proposing of the hypothesis of the possibility of increasing the service life of components of an electric power station fabricated from heat-resistant materials by performing intermediate heat treatments after intervals of time amounting to 25-30% of the design service life. This recommendation must be checked under industrial conditions. In practice the most convenient items for testing are the fittings,

whose service life could be extended significantly by the  
use of intermediate treatment.

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